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ELECTRODEPOSITION OF CHROMIUM
WITH CODEPOSITED PARTICLES

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TECHNICAL REPORT

Linden H. Wagner
and
Richard P. Erickson

May 1972

RESEARCH DIRECTORATE

WEAPONS LABORATORY AT ROCK ISLAND

RESEARCH, DEVELOPMENT AND ENGINEERING DIRECTORATE

U. S. ARMY WEAPONS COMMAND

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13. ABSTRACT

Two techniques were used by the Research Directorate, Weapons Laboratory at Rock Island, for occluding particles of boron carbide, silicon carbide, glass frit, zirconium oxide, aluminum oxide, and diamond dust in standard chromium electrodeposits. The first technique involved the reliance upon the random occlusion of mechanically dispersed particles in the plating solution during electrodeposition of chromium. The second technique involved the use of particles plated with a ferromagnetic coating and a magnetic cathode for magnetic attraction of the particles from the chromium-plating solution to the cathode. Coatings were plated on steel Falex pins and subjected to a modified test procedure with unplated steel V-blocks and VV-L-800 oil. From these data, coefficients of friction and wear properties were determined for each coating. The first technique produced randomly occluded particles which increased the coating coefficient of friction and wear of the Falex blocks. The second technique with magnetically attracted particles produced an unsatisfactory distribution of particles along the surface of the p.. during electroplating. Little variation was noted between the coefficient of friction of the chromium plate with and without particles occluded by this technique. (U) (Wagner, Linden H., and Erickson, Richard P.)

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2. Electroless plating						
3. Chrome plating						
4. Magnetic film						
5. Codeposition of particles						
6. Wear resistance						

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Two techniques were used by the Research Directorate, Weapons Laboratory at Rock Island, for occluding particles of boron carbide, silicon carbide, glass frit, zirconium oxide, aluminum oxide, and diamond dust in standard chromium electrodeposits. The first technique involved the reliance upon the random occlusion of mechanically dispersed particles in the plating solution during electrodeposition of chromium. The second technique involved the use of particles plated with a ferromagnetic coating and a magnetic cathode for magnetic attraction of the particles from the chromium-plating solution to the cathode. Coatings were plated on steel Falex pins and subjected to a modified test procedure with unplated steel V-blocks and VV-L-800 oil. From these data, coefficients of friction and wear properties were determined for each coating. The first technique produced randomly occluded particles which increased the coating coefficient of friction and wear of the Falex blocks. The second technique with magnetically attracted particles produced an unsatisfactory distribution of particles along the surface of the pins during electroplating. Little variation was noted between the coefficient of friction of the chromium plate with and without particles occluded by this technique.

CONTENTS

OBJECTIVE

The objective of this investigation was to establish a method for occluding micron-sized particles in a chromium electrodeposit for use in small arms weapon systems.

INTRODUCTION

Chromium is electrodeposited in the bores of gun tubes for resistance to high temperature and gas erosion to prolong the service life of the tube. To further improve the performance of chromium plate, a program was initiated at Springfield Armory to investigate the application of high-temperature corrosion and erosion-resistant chromium electrodeposits for small caliber weapons and weapon systems. Effort was directed to electrodeposits of chromium in which micron-sized particles of various materials were occluded. These particles were mechanically suspended in the plating bath during electrodeposition of the chromium. Some particles were reported to have become occluded in the chromium matrix. Diamond dispersion coatings were of the greatest interest because of their unusual wear resistance and low coefficient of friction.[1,2,3] Springfield Armory reported these coatings to be superior in wear resistance to the standard chromium electrodeposit. The purpose of this work was to establish methods for occluding particles in a chromium electrodeposit, and to evaluate friction and wear properties.

The work conducted at Springfield Armory was reviewed. The randomly dispersed particles in a chromium solution were shown not to have been attracted to the cathode in a satisfactory manner. Few particles had actually become occluded in the matrix. To increase the number of occluded particles, the particles were placed in a nylon bag fitted around the cathode. This action increased the number of particles in the matrix. However, adhesion of the chromium plate to the basic metal was unsatisfactory.

APPROACH

In establishment of a method for occlusion of particles in a chromium electrodeposit, two factors were considered to be of importance. The particles should be attracted toward the cathode (such as cations are) and the included particles should be large enough in number to influence the properties of the deposit. To direct the particles to the workpiece, coating of the particles with a ferromagnetic film was planned so that these particles would be attracted to a magnetic cathode in the plating bath. Electroless deposition could be used to apply a coating with magnetic properties to the particles. Many chemical reduction solutions for the

electroless deposition of cobalt and nickel are given in current literature.[4,5] These solutions can be used to plate on both metallic and sensitized nonmetallic surfaces.[6] The following nickel and cobalt electroless plating solution formulations were selected.

Nickel Bath[7]

Chemical	Grams per Liter of Distilled Water
Nickel Sulfate ($NiSO_4 \cdot 5H_2O$)	20 - 25
Sodium Citrate ($Na_3C_6H_5O_7 \cdot 2H_2O$)	10 - 15
Sodium Acetate ($NaC_2H_5O_2$)	10 - 15
Ammonium Acid Fluoride (NH_4F_2)	0.35 - 0.50
Sodium Hypophosphite ($NaH_2PO_2 \cdot H_2O$)	15 - 20

Temperature 190 ± 5°F

pH 4-6 adjusted with NaOH

Cobalt Bath[4]

Chemical	Grams per Liter of Distilled Water
Cobalt Chloride ($CoCl_2$)	25 - 40
Ammonium Chloride (NH_4Cl)	25 - 40
Sodium Citrate	70 - 80
Sodium Hypophosphite	15 - 20

Temperature 160 - 180°F

pH 8-10 (adjusted with NH_4OH)

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PROCEDURE

Approximately 15 grams of micron-sized particles were wetted in anhydrous methyl alcohol. Distilled water was added to the slurry, and the particles were allowed to settle so that the alcohol water mixture could be decanted. Particles that required sensitizing[9],[10] before plating were treated for one minute in a solution of 20 grams per liter of stannous chloride and 0.5N hydrochloric acid heated to 160 to 180°F. Particles were filtered, and washed with dilute hydrochloric acid and distilled water. These particles were then added to a solution of 0.1 to 0.2 gram per liter of palladium chloride and 0.2N hydrochloric acid heated to 160 to 180°F. The particles were filtered after one minute, rinsed with distilled water, and wetted with sodium hypophosphite solution before addition to the electroless nickel bath. After the particles were plated for one hour, they were allowed to settle out, and then filtered from the solution. They were then cold-water rinsed, wetted with a sodium hypophosphite solution, and added to an electroless cobalt bath for one hour. The bath was cooled and the particles filtered, after which they were rinsed in methanol and dried at 220°F. To change the phosphate to a phosphide, the particles were heated to 750°F for four hours.[11],[12] This heating was done to increase the magnetic susceptibility and to decrease the solubility of the coating in the chromic acid solution.

To determine the effectiveness of this new method required plating with particles by the random occlusion technique and then plating with particles by the magnetic attraction technique.

Falex pins of SAE 3135 steel were used as specimens to facilitate testing of the coatings. The pins were vapor-degreased in trichloroethylene, and etched for one minute in 10 per cent sulfuric acid at room temperature. After a water rinse and a 30-second etch in 50 per cent hydrochloric acid at room temperature, they were water-rinsed and reverse-etched for 15 seconds in the chromium plating solution at 3.5 volts.

Pins plated with the nonmagnetic fixture were plated two at a time in a vertical position, as shown in Figure 1. The plating solution was a 1200-ml chromic acid bath containing 250g/l chromic acid, 2.5g/l sulfate-ion, and 3-5 per cent trivalent chromium. Two pins, to be used as standards, were plated without any particles present in the bath. Similar
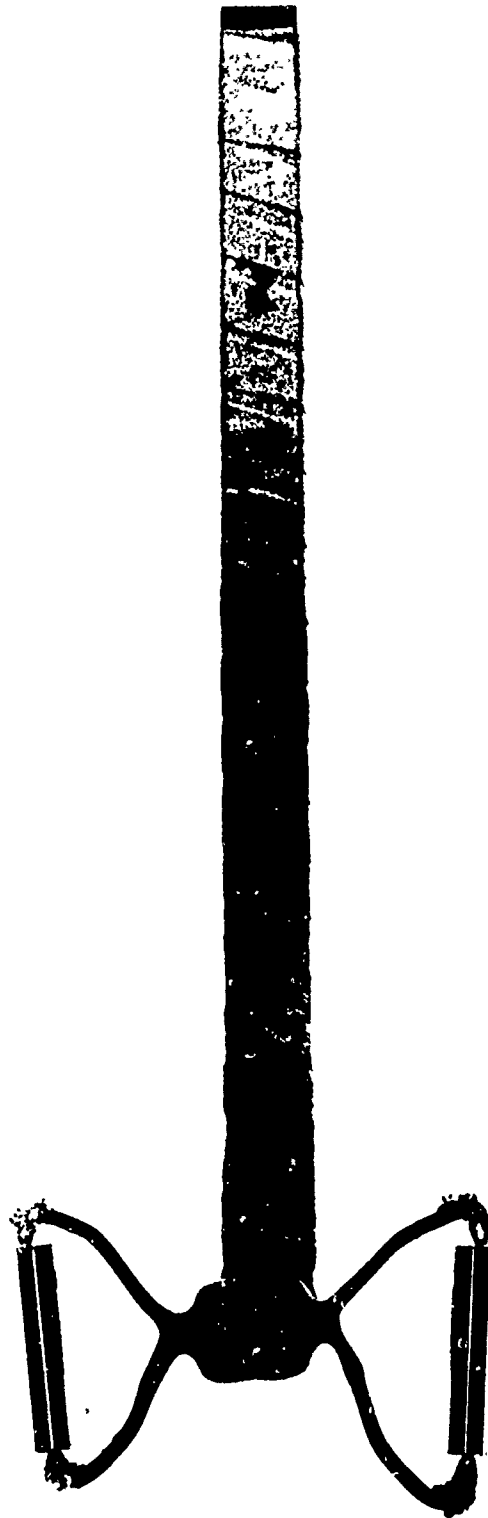
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FIGURE 1   Fixture for Nonmagnetic
Plating of Falex Pins

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pins were plated with 100g/l of 1-5 micron-sized unplated particles present in the bath. The pins were plated for two hours with a current density of 2.5 to 3.0 amps/in^2 at 131 ± 1°F. Constant mechanical stirring was used to keep the particles in suspension.

Pins plated with the magnetic fixture were plated one at a time in a horizontal position. As shown in Figure 2, the pin could be rotated 90° to shift the polar magnetic attraction around the circumference of the pin. The plating bath formulation was the same as given above. Two pins to be used as controls were plated with no particles present in the bath. The other Falex pins were plated with 10g/l of the plated particles present in the bath. All pins were plated for two hours with a current density of 2.5 to 3.0 amps/in^2 at 131°F. Constant mechanical stirring was used to keep the particles in suspension. The electromagnet was turned on during the entire plating time, and the pin was rotated on its axis every ten minutes.

After the pin was plated, it was measured with a micrometer to determine coating thickness; weighed and visually inspected for brightness, uniformity, and particle inclusion.

Falex tests were conducted on the standard chromium-plated pins with uncoated V blocks of SAE 1137 steel and VV-L-800 preservative oil. Because of the superior wear properties of the chromium deposits in this system, the load schedule was made more stringent to include a break-in period of three minutes at a 250-pound load and a load increase of 250 pounds every five minutes thereafter until failure or a maximum of 3500 pounds was applied. The torque increase was noted for each increase in the applied load. From this, the coefficient of friction was calculated according to the following equation.[13]

$$\text{Coefficient of friction} = \frac{\text{Torque (in-lb)}}{\sin \theta \times \text{Load (lb)} \times \text{Radius of pin (in)}}$$

where $\theta \cong 45°$ ($\sin \theta = 0.707$) and is half the angle subtended by the chuck jaws. This geometric factor accounts for the fact that the applied load must be modified to get the normal, effective load. The coefficient of friction is "effective" only because one-dimensional contact along the length of the pin is made instead of two-dimensional contacts over the entire cylindrical surface area. V blocks and pins were weighed before and after each test to determine the relative wear. The pins were cross-sectioned and mounted for polishing and metallographic examination.
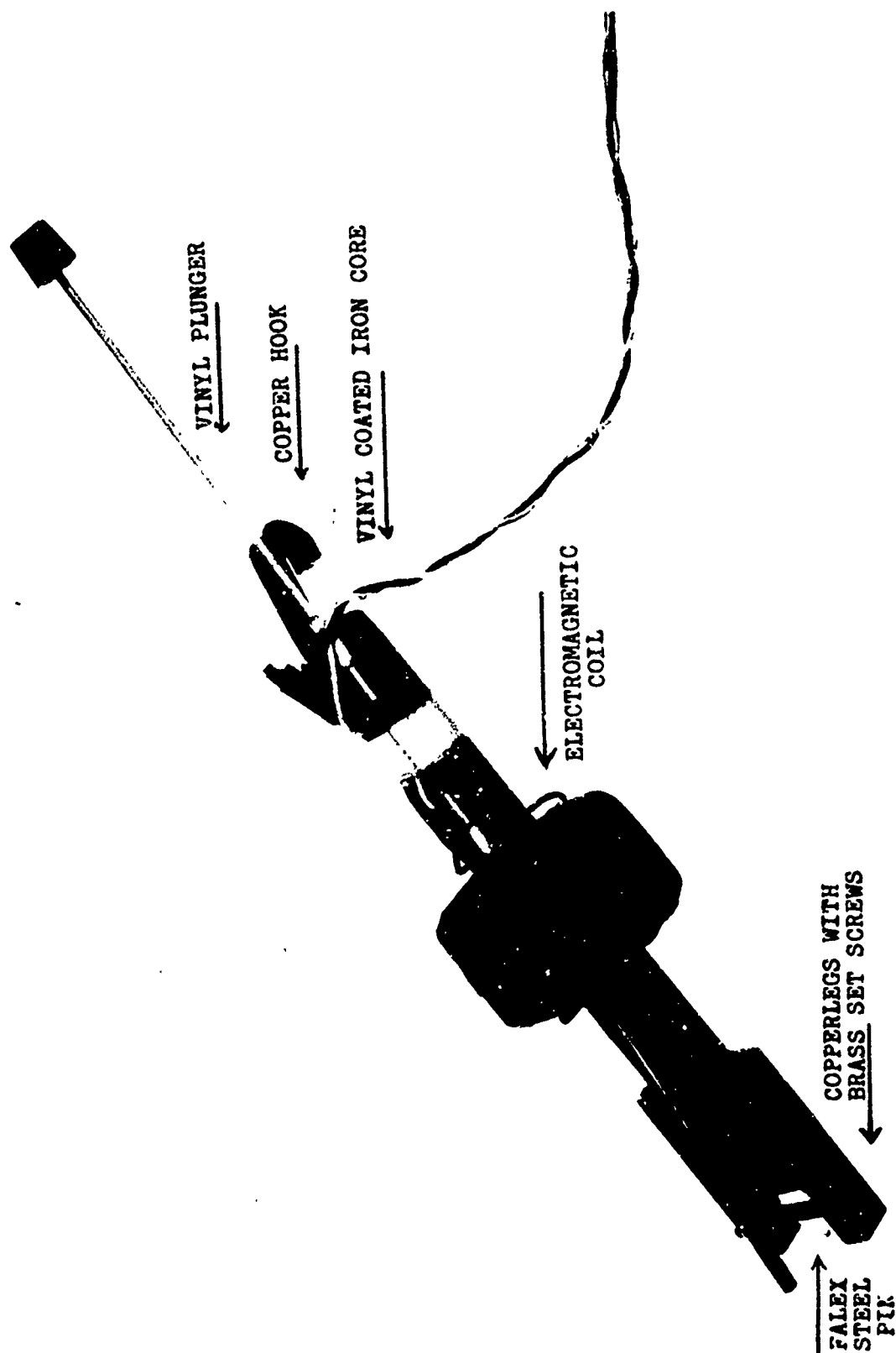
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VINYL PLUNGER

COPPER HOOK

VINYL COATED IRON CORE

ELECTROMAGNETIC COIL

COPPER LEGS WITH BRASS SET SCREWS

FALEX STEEL PIN

FIGURE 2   Magnetic Cathode with Movable Falex Pin

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RESULTS

The results of this investigation to improve chromium electrodeposits by means of particle occlusion have been divided into the random occlusion and the magnetic attraction techniques. Load-torque relationships from Falex tests are tabulated in Tables I and IV. Selected results have been represented in Figures 3 and 5. The calculated coefficients of friction are listed in Tables II and V. Selected results are plotted in Figures 4 and 6. A summary of weight losses is given in Table III for the random occlusion technique and in Table VI for the magnetic attraction technique.

The torque-load relationships, shown in Table I, represent the average torque maintained during the period for each load. Possible significant deviations in the wear characteristics of chromium electrodeposits with randomly occluded particles from those of the standard chromium might be indicated by the lower torque readings in the load range of 750 to 1750 pounds. However, final torque readings tended to merge toward the maximum load of 3500 pounds as shown in Figure 3; this indicates either unsatisfactory performance of the occluded particles or too few occlusions to affect the wear characteristics at the higher loads. Examinations of cross-sectioned areas tended to support the latter (Figure 7), but whether a totally representative picture of the occlusions can be obtained in this manner is not certain. Calculated coefficients of friction as shown in Table II, are in relatively close agreement where loads over 1000 pounds have been applied. From this point, a gradual decrease is noted, as shown in Figure 4, to a value of approximately 0.25 at a 3500-pound load. For loads under 750 pounds, standard chromium plate offers the lowest coefficient of friction for any of the coatings.

Wear properties based on weight loss of pins and blocks after testing are given in Table III with the maximum torque and total time in test. When a pin failure occurred, the resulting reduction in test time and corresponding weight loss was noted. Because of the insignificant weight loss of the pins, this method was unsatisfactory for comparison of the actual wear properties of the coatings. However, block wear was uniform, by which an appreciable weight loss was shown that might be considered an indication of the abrasive qualities of the coatings.

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TABLE I

AVERAGE TORQUE ON FALEX PIN PLATED IN CHROMIUM BATH WITH DISPERSION OCCLUDED PARTICLES (INCH-POUNDS)

Dispersion Occluded Particles (100 gms/liter)	250	500	750	1000	1250	1500	1750	2000	2250	2500	2750	3000	3250	3500
Chromium Plated Pin (Control)	11.5	17.5	24.0	36.5	40.0	44.5	50.0	54.5	59.0	63.0	69.0	72.0	75.0	78.3
Boron Carbide	14.0	20.0	24.0	35.0	39.0	42.0	47.0	53.0	59.0	63.0	67.0	70.5	72.0	75.0
Silicon Carbide	15.0	21.0	28.0	35.5	39.0	43.0	48.0	60.0	66.0	68.0	71.5	71.0	77.0	83.0
Zirconium Oxide	12.0	17.0	22.0	31.0	36.5	40.0	44.0	50.0	61.5	63.0	63.5	68.0	73.5	77.0
Glass Frit	11.5	16.5	22.5	30.0	36.0	40.0	43.5	55.0	63.0	66.0	66.0	68.0	72.0	77.0
Aluminum Oxide	13.0	17.5	22.5	29.0	35.0	40.0	43.5	53.0	61.0	64.5	62.5	68.0	72.0	75.5

(Load in Pounds)

TABLE II

COEFFICIENTS OF FRICTION OF CHROMIUM ELECTRODEPOSITS WITH DISPERSED OCCLUDED PARTICLES

Type of Particles	Load in Pounds													
	250	500	750	1000	1250	1500	1750	2000	2250	2500	2750	3000	3250	3500
None - Standard Chromium	.52	.40	.365	.415	.36	.33	.33	.31	.31	.295	.285	.275	.26	.25
Boron Carbide	.64	.45	.365	.40	.36	.325	.315	.30	.295	.29	.28	.27	.25	.24
Silicon Carbide	.68	.49	.425	.405	.36	.32	.32	.34	.33	.31	.29	.27	.27	.27
Zirconium Oxide	.55	.39	.34	.34	.33	.30	.28	.28	.31	.29	.26	.25	.26	.25
Glass Frit	.56	.37	.34	.34	.33	.30	.26	.30	.31	.29	.26	.26	.25	.25
Aluminum Oxide	.64	.41	.34	.33	.32	.30	.28	.29	.31	.28	.25	.25	.25	.25

TABLE III

FALEX TESTS OF CHROMIUM ELECTRODEPOSITS
WITH RANDOMLY OCCLUDED PARTICLES

Dispersion Occluded Particle	Loss of Wt. of Falex Pin (grams)	Loss of Wt. of V-Blocks (grams)	Max. Load (lbs)	Max. Torque (in-lb)
Chromium Plated Control	-	0.36	3500	76
Chromium Plated Control	0.001	0.15	3500	88
Boron Carbide	Shear pin broke	0.30	3500	85
Boron Carbide	0.003	0.12	3500	67
Silicon Carbide	Shear pin broke	0.49	3500	85
Silicon Carbide	Falex pin broke	0.12	2750	75
Zirconium Oxide	0.004	0.27	3500	78
Zirconium Oxide	0.002	0.23	3500	76
Glass Frit	0.001	0.33	3500	77
Glass Frit	0.001	0.31	3500	78
Aluminum Oxide	0.001	0.23	3500	72
Aluminum Oxide	0.001	0.28	3500	78

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TABLE IV

AVERAGE TORQUE ON FALEX PIN PLATED IN CHROMIUM BATH
WITH MAGNETICALLY ATTRACTED PARTICLES (INCH-POUNDS)

Magnetically Attracted Particles (10 gms/liter)	Load in Pounds													
	250	500	750	1000	1250	1500	1750	2000	2250	2500	2750	3000	3250	3500
Chromium Plated Pin (Control)	10.5	16.0	20.0	27.5	35.0	40.0	43.5	48.0	52.0	59.5	60.5	63.0	66.5	69.0
Boron Carbide	12.5	15.5	21.5	27.0	32.0	37.0	41.5	45.5	51.0	57.0	63.0	66.0	67.0	71.0
Silicon Carbide	12.5	14.5	18.5	24.5	31.0	37.0	40.5	44.0	50.0	58.0	60.0	63.0	65.0	68.0
Zirconium Oxide	11.0	14.0	18.0	25.0	31.0	37.0	39.5	42.5	51.0	58.5	60.5	61.0	64.5	67.5
Glass Frit	11.5	16.5	20.0	28.0	35.0	39.0	42.5	47.0	56.0	63.0	65.0	66.0	66.0	67.0
Aluminum Oxide	12.5	15.5	19.0	29.0	33.5	37.8	42.5	45.0	51.5	58.0	59.5	61.5	63.5	67.5
Diamond Dust	11.5	15.5	20.0	26.5	36.5	39.5	42.0	47.0	55.0	63.0	63.5	60.0	62.0	65.0

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TABLE V

COEFFICIENTS OF FRICTION OF CHROMIUM ELECTRODEPOSITS WITH MAGNETICALLY ATTRACTED PARTICLES

Magnetically Attracted Particles	Load in Pounds													
	250	500	750	1000	1250	1500	1750	2000	2250	2500	2750	3000	3250	3500
None - Standard Chromium	.50	.36	.30	.31	.32	.30	.28	.27	.26	.27	.25	.24	.235	.23
Boron Carbide	.55	.34	.32	.31	.29	.28	.27	.26	.26	.26	.26	.25	.24	.23
Silicon Carbide	.55	.32	.29	.28	.28	.28	.28	.26	.25	.26	.25	.24	.23	.22
Zirconium Oxide	.50	.33	.27	.28	.28	.28	.27	.24	.26	.27	.25	.23	.23	.22
Glass Frit	.52	.38	.30	.32	.32	.29	.28	.27	.28	.30	.27	.25	.23	.21
Aluminum Oxide	.55	.35	.29	.33	.30	.29	.27	.26	.26	.26	.25	.23	.22	.22
Diamond Dust	.46	.36	.30	.31	.32	.295	.27	.27	.26	.29	.27	.23	.22	.21

TABLE VI

FALEX TESTS OF CHROMIUM ELECTRODEPOSITS
WITH MAGNETICALLY ATTRACTED PARTICLES

Magnetically Attracted Particle	Loss of Wt. of Falex Pin (grams)	Loss of Wt. of V-Blocks (grams)	Max. Load (lbs)	Max. Torque (lb-in)
Chromium Plated Control Pin	None	0.13	3500	69
Boron Carbide	None	0.39	3500	76
Boron Carbide	None	0.31	3500	69
Silicon Carbide	-	0.27	3500	67
Silicon Carbide	-	0.14	3500	68
Zirconium Oxide	0.001	0.16	3500	68
Glass Frit	0.003	0.09	3500	67
Aluminum Oxide	0.001	0.14	3500	68
Diamond Dust	0.001	0.11	3500	65

None of the tests failed

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FIGURE 3

Graph showing torque-load relationship of Falex pins plated with chromium and dispersion occluded particles.

Code:
O Standard Chromium
□ Chromium + Boron Carbide
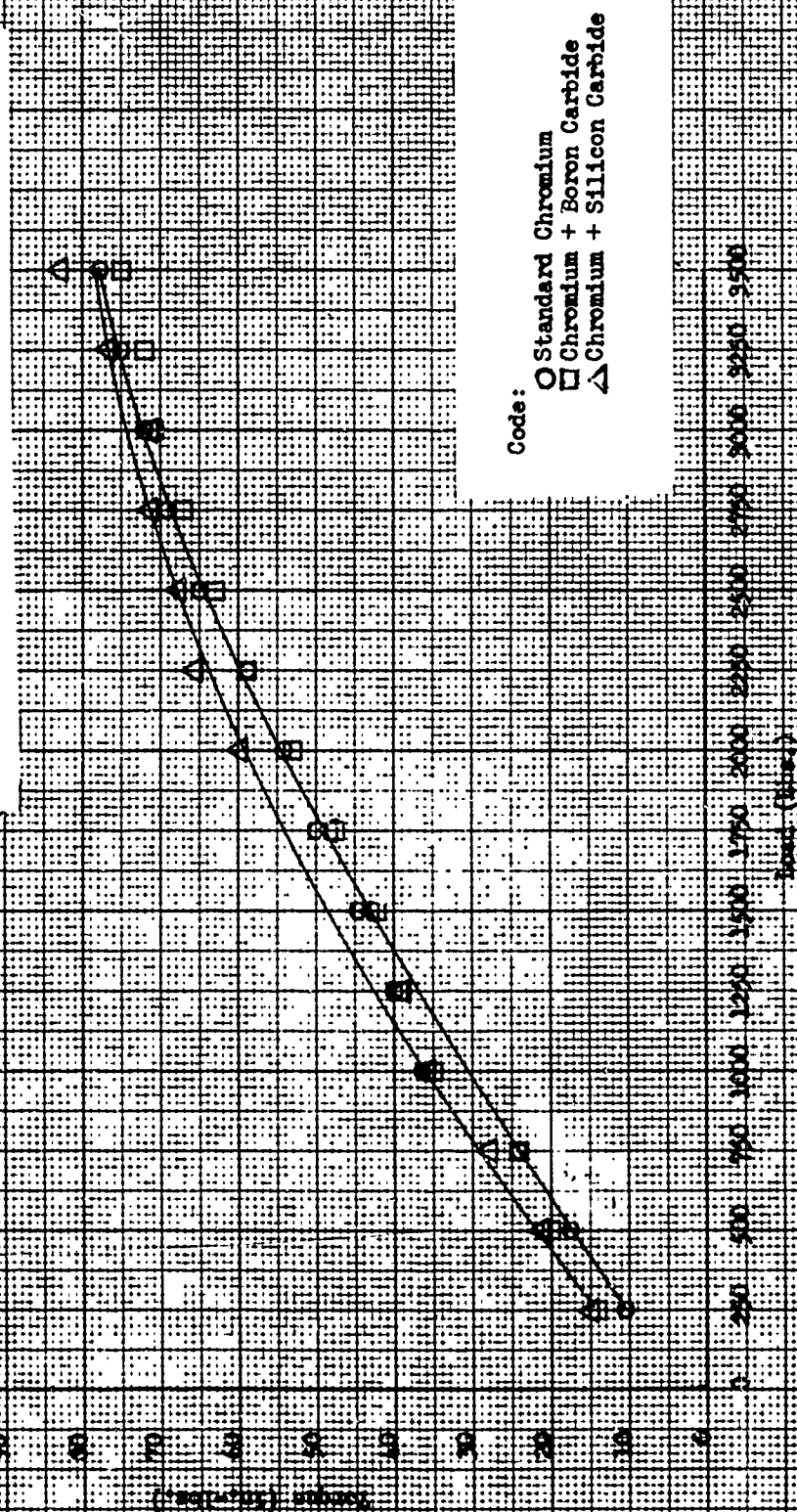△ Chromium + Silicon Carbide

FIGURE 4

Graph showing coefficient of friction of chromium
electrodeposits with dispersion occluded particles.

Code:
○ Standard Chromium
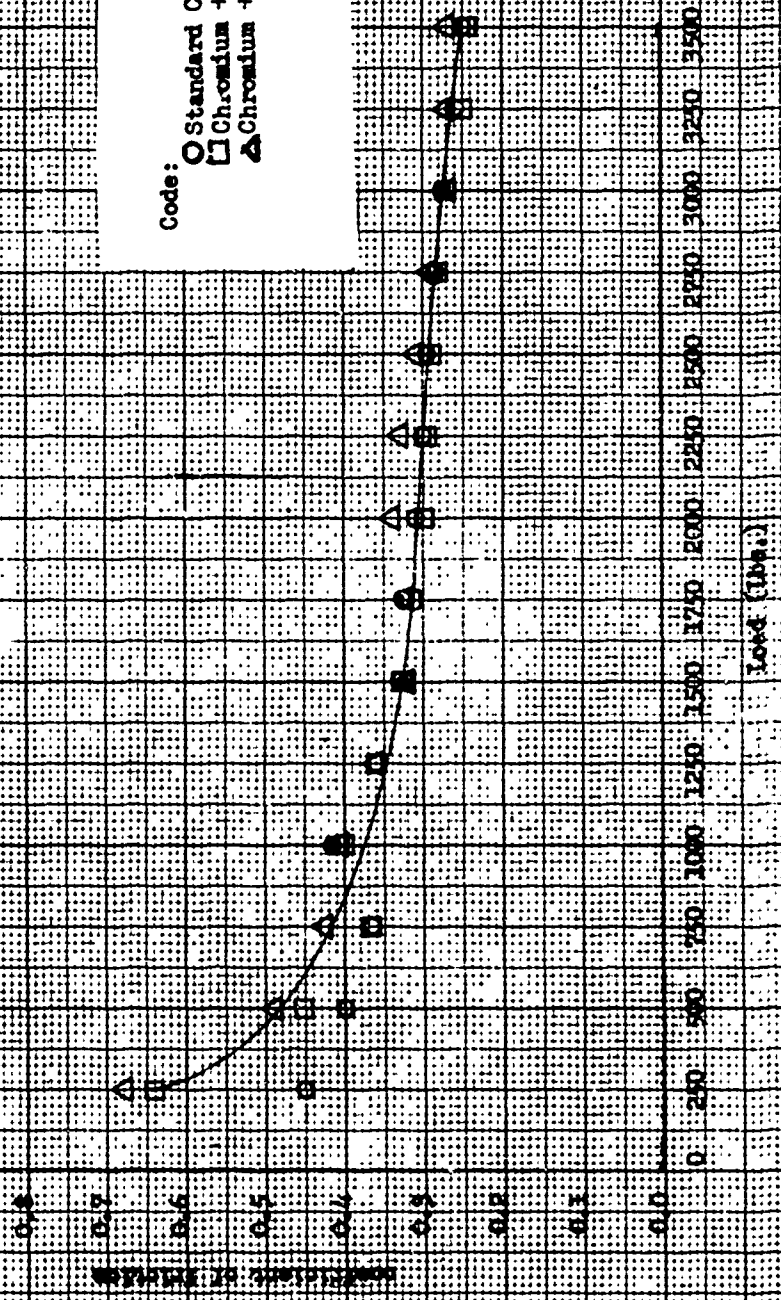□ Chromium + Boron Carbide
△ Chromium + Silicon Carbide

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FIGURE 5

Graph showing torque-load relationships of Falex pins plated with chromium and magnetically attracted particles.

Code:
⊙ Standard Chromium
☐ Chromium + Boron Carbide
△ Chromium + Silicon Carbide
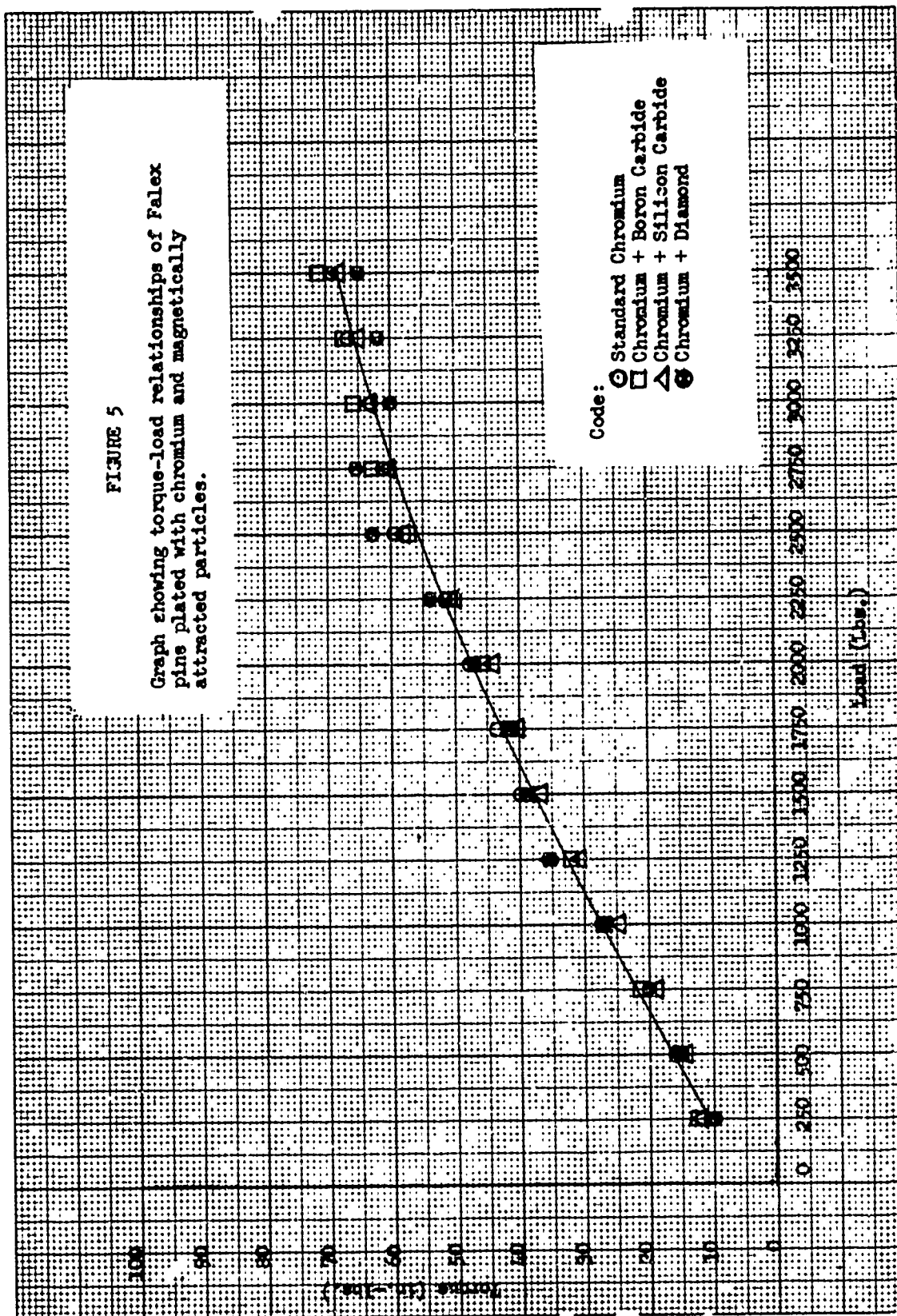⬤ Chromium + Diamond

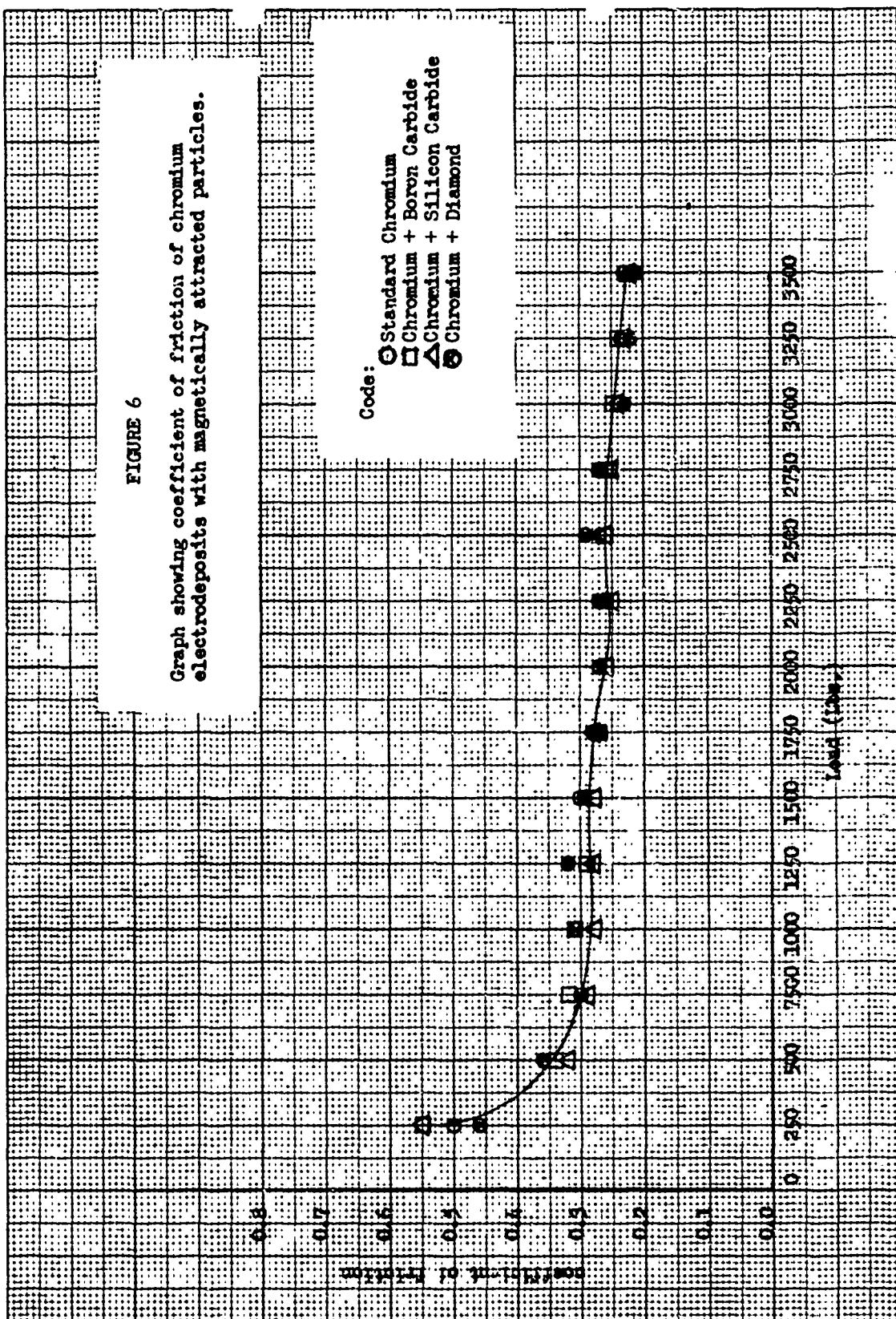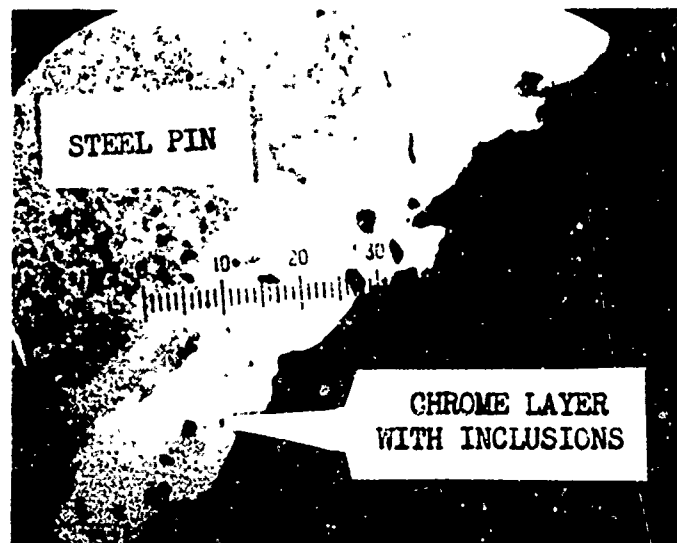Load (Lbs.)

Torque (in.-lbs.)

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FIGURE 6

Graph showing coefficient of friction of chromium electrodeposits with magnetically attracted particles.

Code:
⊙ Standard Chromium
□ Chromium + Boron Carbide
△ Chromium + Silicon Carbide
⊕ Chromium + Diamond

17

Labels within figure: STEEL PIN; CHROME LAYER WITH INCLUSIONS

FIGURE 7   Cross Section of Plated Pin
with Typical Scattering of Inclusions

400X

The performance of the standard chromium electrodeposits versus the deposits with the magnetically attracted particles (Table IV and Figure 5) is almost identical. Only slight variations were noted during the initial break-in period of a 250-pound load, followed by an almost linear increase in torque relative to the increasing load up to 2250 pounds. From the results of a comparison of the calculated co-efficients of friction, a close agreement is shown between the specimens. The loss in weight of the Falex pins (Table VI) was slight; not any of the pins failed during the test period. Wear was considerable with respect to the coating containing glass frit. Although coating containing diamond dust also appeared to have a low level of abrasion, weight loss was less than that of the coating containing glass frit.

DISCUSSION

The use of large concentrations of particles in the chromium plating bath seemed to result in only randomly dispersed particles throughout the chromium electrodeposit. After the 3-minute break-in period at a 250-pound load, the torque measurements recorded for the different coatings varied up to 2000 pounds, with the standard chromium slightly higher than that of the coatings. Calculated coefficients of friction for the coatings were approximate at a load of 2000 pounds, and the standard chromium deposit was again slightly above the average up to this point. Of the twelve pins tested in this series, three failures were noted before the completion of the test schedule.

The second technique, involving the use of magnetically attracted particles, was unsatisfactory because the particles would line up along the longitudinal magnetic pole of the pin top and bottom. Even with the rotation of the pin effecting a shift of the magnetic poles, a uniform attraction to the surface of the pin was impossible. Another problem encountered with this technique was the dissolving of the metallic coatings on the particles in the chromic acid solution. After approximately four hours of exposure, the particles seemed to lose most of the magnetic characteristics. This analysis is supported by Table IV since less variation in the Falex test is shown between the standard chromium and the chromium with magnetically attracted particles than in the random occlusion used in the first technique. No test failures were noted in this second series, probably because of the more uniform chromium electrodeposits produced by the more favorable geometry of the second cathode, anode relationships. This would account for the superior test results of the second standard chromium deposit

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compared with the first as seen in Figures 5 and 3, respectively.

The weight loss of the V blocks during testing (Tables III and VI) seems significant enough to represent the abrasive properties of the chromium electrodeposit. However, because the chromium deposit was substantially harder than the surface of the blocks, no significant weight loss was recorded. The greater weight losses (Table III) are representative of the greater number of particles occluded in the deposit.

The electroless reduction plating of nickel and cobalt is ideally suitable for plating these micron-sized particles. Particles that cannot be plated directly can be activated by a dip in stannous chloride and then a dip in palladium chloride. Each batch of plated particles was tested with a magnet to ensure attraction of them to the cathode during electrodeposition. An additional check of the particles two hours after immersion of them into the bath was also used to determine whether a sufficient number of particles had retained ferromagnetic properties during the plating period.

CONCLUSIONS

1. The random presence of minute particles in a chromium plating bath does not necessarily produce a satisfactory electroplate with a significant number of occluded particles in the matrix for two reasons: First, the agitation necessary to keep the particles suspended in the bath tends to sweep the particles from the cathode surface. Secondly, the formation of hydrogen gas at the cathode during electrodeposition lifts the particles away from the surface.

2. Magnetic particles dispersed in a chromium plating bath are attracted to the magnetic cathode in the system used in this work.

3. The magnetic attraction between the particles, and the cathode does aid in the occlusion of these particles in the electrodeposited chromium in this system. However, the distribution is nonuniform because of the orientation of the magnetic field.

4. Changing the phosphate to a phosphide by heating the nickel-cobalt coatings decreases the solubility of these coatings in chromic-acid plating solutions.

5. Particles of diamond dust, boron carbide, silicon carbide, aluminum oxide, and zirconium oxide codeposited in a chromium electroplate increased the abrasive properties of the deposit.

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RECOMMENDATIONS

1. Further work should be performed to determine whether an increase in particle size will improve the friction and wear properties.

2. The use of borane reducing agents for the electroless plating of nickel and cobalt magnetic films should be investigated.

3. Other methods for attracting particles to the cathode should be investigated.

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